Indian J. Phys. **52A**, 50–54 (1978)

Low angle boundaries in magnesium orthosilicate single crystals

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(*Received* 29 *September* 1976, *revised* 4 *January* 1977)

Synthetic magnesium orthosilicate (forsterite) crystals have been chemically etched in the melt of potassium hydroxide at 400°C. Rows of equally spaced etch pits have been observed. By etching matched cleavage faces, correspondence has been observed for intersecting boundaries. Correspondence has also been established on the two sides of a thin plate 1mm thick. It is observed that the density of pits in all the corresponding rows on matched cleavage faces and on the two sides of a thin plate is the same. It is established that in the intersecting boundaries, the density of pits in one branch is equal to the sum of densities in the remaining two branches. The implications are discussed.

1. Introduction

The structure of real crystals is usually different from that of the ideal ones in which the lattice planes have the same orientation throughout the crystal. Microscopic studies as well as examination by the X-ray diffraction have shown that real crystals are composed of small blocks with slightly different orientations which give rise to what are called low-angle grain boundaries. Burgers (1940) and Bragg (1940) have shown that low-angle tilt boundaries consist of arrays of edge dislocations. Read & Shickley (1950) predicated certain physical properties such as grain boundary energy on the basis of the assumed dislocation model. Vogel *et al* (1953) have reported that the row of equally spaced etch pits in germanium crystals represented an array of edge dislocations, formed owing to a small-angle tilt boundary. In the case of intersecting boundaries consisting of edge dislocations having a single Burgers vector, it is shown that $n_a+n_b = n_c$, where n_a, n_b and n_c are the densities of dislocations in the three branhes. The density of dislocations may be counted by counting the etch-pits. Low-angle grains boundaries have been reported, as for example, by Amelinckx (1954) on NaCl, Wagner & Chalmers (1960) on germanium, Patel & Koshy (1968) on barite. In this paper, we describe small-angle boundaries observed on laboratory grown single crystals of magnesium orthosilicate.

2. Experimental and Observations

For experimental purposes, synthetic forsterite crystals obtained from Oak Ridge National Laboratory, U.S.A., were cleaved along (010) planes and freshly cleaved faces were etched in the melt of potassium hydroxide at 400°C for a few seconds They were then examined by optical microscopy after depositing thin silver films on them They were also examined in an electron microscope by preparing a single-stage carbon replica as reported by Patel & Patel (1968) Figure 1 illustrates the etch pattern in which a row of elongated etch pits fully resolved is clearly seen. The structure of the pits in the row is fully resolved in the electron micrograph shown in figure 2 Assuming that the row represents a low-

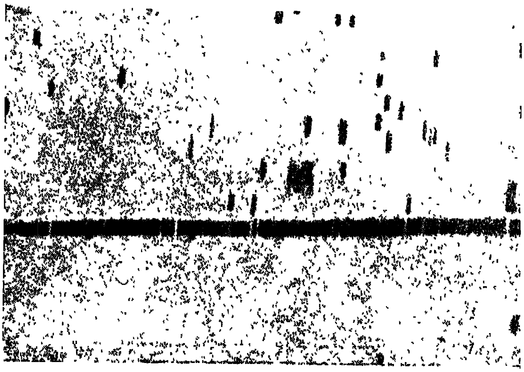


Fig. 1 Low-angle tilt boundary on a (010) plane (×225)

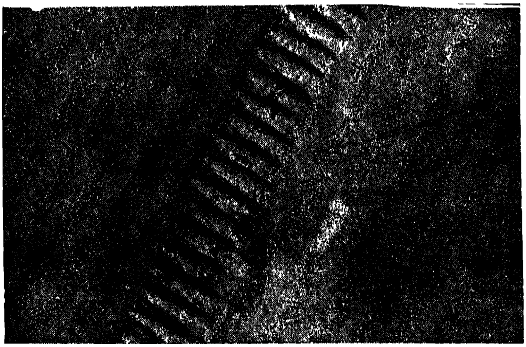
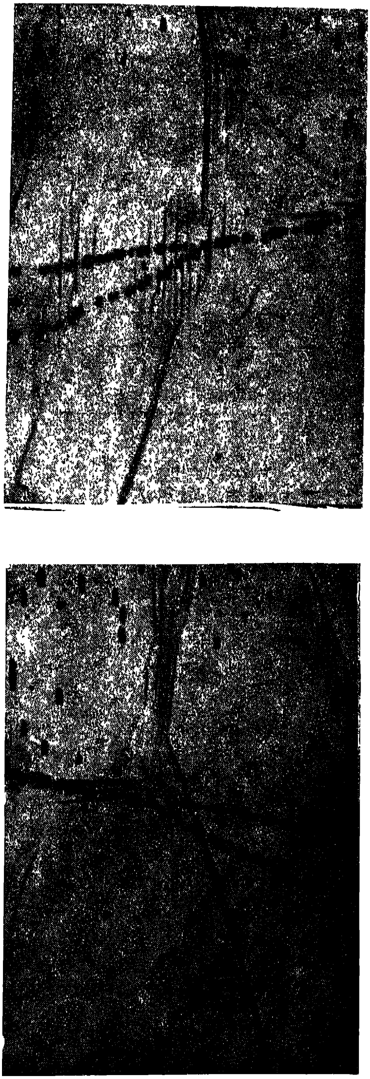


Fig. Electron micrographs of a row of pits (×4 000)

angle tilt boundary, the tilt is calculated to be 24 seconds To investigate how far into the crystal such boundaries extend, a thin flake of about 1mm thickness

Figs. 3(a) & (b) Low-angle tilt boundaries matched on the opposite sides of a thin flake. ($\times 250$)



Figs. 4(a) & (b). Three intersecting rows of pits on the oppositely matched face. ($\times 250$)

was cleaved from a crystal and then etched, figures 3(a) and 3(b) represent the etch patterns on the two sides of the flake. It is noteworthy that the etch patterns match on the two sides of a flake, clearly indicating that the low-angle tilt boundaries run into the body of the crystal

In order to ascertain whether such rows of pits represent tilt boundaries, some intersecting boundaries were sought for Matched cleavage faces shown after etching. Figures 4(a) and 4(b) show examples of grain boundaries in which three boundaries meet at a point The densities of the pits in each row have been calculated and are given in table 1

Table 1. Pit density in Pits/μm for intersecting boundaries on matched pairs

	One Face	Oppositely matched face		
Branch	n_b	n'_a	n'_b	n'_c
No. of pits/μm	0·280	0·718	0 280	0·434

It may be noted that .

1. The density of pits in each branch on one cleavage face is the same as the density of pits on the corresponding branch on the matched face.
2. t the junction of three boundaries on both the faces, the relation $n_a = n_b + n_c$ is satisfied.
3. In addition to the matching of grain boundaries, there is one-to-one correspondence in the size and position of the individual pits on them

3. Conclusions

It is quite clear from the work described above that the rows of pits observed in magnesium orthosilicate single crystals represent low-angle tilt boundaries, which consist of rows of edge dislocations and that the pits in the boundaries are the dislocation etch pits.

Acknowledgment

The authors wish to record their sincere thanks to Prof A R Patel for his keen interest in the study One of the authors (J V.) wishes to thank the University Grants Commission, New Delhi, for financial support.

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